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Space systems $\frac{3}{4}$ Safety and compatibility of materials – Part 5: Test method for determining the reactivity of system/component materials with aerospace hypergolic propellants

*Systèmes spatiaux — Sécurité et compatibilité des matériaux - Partie 5: Methode d'essai pour
determination de la reactivité des matériaux de système ou pièces avec propellants hyper-
goliques spatiaux*



Reference number
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Contents

1	Scope	1
2	Normative references.....	1
3	Definitions.....	1
4	Fluid transfer, storage, and flight systems.....	2
4.1	General	2
4.1.1	Procedure	2
4.1.2	Safety.....	2
4.2	Test criteria	2
4.2.1	Screening test	2
4.2.2	Immersion test.....	2
4.2.3	Posttest analysis.....	2
4.3	Sample.....	2
4.3.1	Receiving inspection.....	2
4.3.2	Sample preparation	2
4.3.3	Sample cleaning.....	3
4.3.4	Sample inspection	3
4.4	Test system.....	3
4.4.1	Screening test	3
4.4.2	Immersion test.....	3
4.5	Test procedure	3
4.5.1	Pretest procedure.....	3
4.5.2	Test procedure	3
4.5.2.1	Screening test	3
4.5.2.2	Immersion test.....	5
4.5.3	Posttest analysis.....	6
4.6	Precision	6
	Table 1 – Material identification.....	4
	Table 2 – Screening test report form.....	7
	Table 3 – Posttest analysis test report form (2 sheets).....	8
	Figure 1 – Immersion test system	5

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and nongovernmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 14624-5 was prepared by Technical Committee ISO/TC 20, Aircraft and Space Vehicles; Sub-Committee SC 14, Space Systems and Operations.

ISO CD 14624 consists of the following parts, under the general title Space systems – Safety and compatibility of materials:

Part 1: Test method for upward flammability of materials

Part 2: Test method for determination of electrical wire insulation and accessory flammability

Part 3: Test method for determination of offgassed products from materials and assembled articles

Part 4: Test method for upward flammability of materials in gaseous oxygen and oxygen-enriched environments

Part 5: Test method for determining the reactivity of system/component materials with aerospace hypergolic propellants

Part 6: Test method for determining the reactivity of processing materials with aerospace fluids

Part 7: Test method for determining the permeability and penetration of materials to aerospace fluids

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Introduction

This purpose of this International Standard is to identify changes resulting from exposure of a material to an aerospace fluid that renders either the material or the fluid unsuitable for use.

All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

Space systems ¾ Safety and compatibility of materials – Part 5: Test method for determining the reactivity of system/component materials with aerospace hypergolic propellants

1 Scope

This International Standard describes test equipment and techniques used to identify interactions resulting from exposure of a material to an aerospace fluid.

This International Standard may be used to determine the reactivity of system and component materials with aerospace fluids. This International Standard is applicable for determining interactive reactions between propellants and materials used in the design, construction, and operation of propellants storage, transfer, and flight systems. While this procedure is an excellent quick screen test for long-term propellant compatibility, it is semi-qualitative, and (if exposures will exceed 12 months) long-term tests should be used to quantify degradation as a function of time under use conditions.

2 Normative references

The following normative references contain provisions that, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid.

ISO 4954:1993, Steels for cold heading and cold extruding.

ISO 14951-3:1999, Space systems — Fluid characteristics — Part 3: Nitrogen.

ISO 14951-5:1999, Space systems – Fluid characteristics – Part 5: Nitrogen tetroxide propellant.

ISO 14951-6:1999, Space systems – Fluids characteristics – Part 6: Monomethylhydrazine propellant.

ISO 14951-7:1999, Space systems – Fluid characteristics – Part 7: Hydrazine propellant.

ISO 14951-10:1999, Space systems – Fluid characteristics – Part 10: Water.

3 Definitions

For the purposes of this International Standard, the following terms and definitions shall apply:

3.1

degradation

an adverse physical or chemical change in a substance

3.2

immersion test

a test in which the fluid covers the entire sample for the duration of the test

3.3

propellants

fluids, such as hydrazine and monomethylhydrazine, and oxidizers usually used for space projects

3.4

reaction

a chemical change in which a substance decomposes, combines with other substances, or interchanges constituents with other substances

4 Fluid transfer, storage, and flight systems

4.1 General

4.1.1 Procedure

This procedure is applicable for determining interactive reactions between propellants and materials used in the design, construction, and operation of propellant storage, transfer, and flight systems. The sample is immersed in the test fluid for 48 hours at the maximum system temperature or 70 degrees Celsius (°C), whichever is higher. This accelerated test provides semi-qualitative information. Tests used to evaluate the long-term interaction of materials with reactive fluids should be conducted for a period of time no less than that of the anticipated time of use.

4.1.2 Safety

The proper safety equipment must be worn by the technician performing the test. A face shield, gloves, and a laboratory coat or apron shall be worn when handling the test fluids. The laboratory conducting the tests should have a detailed emergency plan in the event of a runaway reaction.

4.2 Test criteria

4.2.1 Screening test

Exposure of the material (Screening Test) to the fluid for 2 hours at ambient temperature and pressure shall not visibly change either the material or the fluid.

4.2.2 Immersion test

The sample immersed in the test fluid for 48 hours at test temperature shall not cause a pressure rate increase that is 1,5 times more than the pressure rate increase that is caused by ISO 4954 stainless steel when exposed to the identical fluid at those conditions. The standard test temperature for the propellant hydrazines is 70 °C. This temperature should be used when the intent of the test is ranking of materials or comparison to literature information. Other temperatures may be used to test materials for specific applications. For other fluids, the standard test temperature will depend upon the vapor pressure of that fluid; for example, the standard temperature for nitrogen tetroxide is 21 °C.

For fluids that do not decompose into gaseous products at the test temperature (for example, nitrogen tetroxide), a pressure increase greater than the vapor pressure of the fluid after exposure to polytetrafluoroethylene (for nonmetals) or ISO 4954 stainless steel (for metals) must not occur.

4.2.3 Posttest analysis

After the sample has been exposed, decontaminated, and dried, no visible change in color or texture of the material or test fluid shall be apparent. In addition, the sample weight change must not be greater than $\pm 2\%$.

The following changes in the fluid must also not occur: (1) the mass of impurities in the fluid after exposure to the material must not be greater than twice the mass of impurities in the identical fluid after exposure to polytetrafluoroethylene (for nonmetals) or ISO 4954 stainless steel (for metals), and (2) halide (F^- , Cl^- , Br^-) concentrations in the fluid after exposure to the material must not exceed the appropriate ISO specification for the fluid purity.

4.3 Sample

4.3.1 Receiving inspection

When received, the test material must be accompanied by proper identification. The minimum information required is the manufacturer, trade name, composition, specification, generic name, and batch/lot number (if known). A visual inspection shall be performed and any anomalies shall be noted. A suitable material identification form is shown in Table 1.

4.3.2 Sample preparation

The sample should be tested in the intended use form (such as sheets or foams) and in the as-received thickness. Samples for the screening test should weigh $\leq 0,25$ gram (g). Samples for the immersion test should have a surface area of 25 ± 10 square millimetres (mm^2).

4.3.3 Sample cleaning

Samples shall be cleaned and dried to the end-use specifications. Contamination on the surfaces of solid, nonporous samples shall be removed by washing with deionized water and mild detergent, rinsing with deionized water, and drying with filtered, gaseous nitrogen. Particulate on the surfaces of solid, porous samples shall be removed with filtered, gaseous nitrogen meeting the requirements of ISO 14951-3.

4.3.4 Sample inspection

The cleaned sample shall be inspected to ensure it is at the specified worst-case thickness. Flaws and any residual contamination shall be noted. If the flaws result from sample preparation at the test facility, new samples shall be prepared. Samples with flaws that inordinately increase the surface area to bulk mass ratios should not be tested. Samples shall be weighed and individually identified.

4.4 Test system

4.4.1 Screening test

The test system for the screening test shall consist of a glass beaker.

4.4.2 Immersion test

The test system for the immersion shall consist of one reference and one sample chamber and temperature and pressure measuring devices (see Figure 2). Differential pressure transducers may be used for fluids, such as hydrazine and monomethylhydrazine, that decompose into gases at the test temperature. Absolute pressure transducers (on the sample and reference sides of the test system) may be used for those fluids that do not decompose into gases or undergo wide pressure fluctuations. Recommended analytical instruments for the posttest analyses include a differential scanning calorimeter, gas chromatography, gas chromatography/mass spectrograph, atomic absorption spectrophotometer, inductively coupled plasma optical spectrometer, inductively coupled plasma/mass spectrometer, ion chromatography, and high-performance liquid chromatography.

4.5 Test procedure

4.5.1 Pretest procedure

The test system shall be clean, and all measuring devices shall be in current calibration. The pretest procedure shall be as follows:

- a) Analyze the fluid to be used in testing for impurities
- b) Verify the fluid meets the required use specifications before being exposed to the samples.
- c) Record all pertinent information for the test, such as sample identification and pretest information about the sample and fluid.
- d) Clean and dry the test and reference samples.
- e) Photograph the samples.

4.5.2 Test procedure

4.5.2.1 Screening test

The screening test shall be as follows:

- a) Place a 0,25 g test sample in the glass beaker.
- b) Apply 10 cubic millimetres (mm³) one drop at a time to the test sample at ambient temperature and pressure.
- c) Wait 2 hours and then examine the sample and fluid visually for obvious changes caused by the exposure.
- d) A suitable screening test report form is shown in Figure 2

Table 1 - Material identification

Test Material

Manufacturer _____

Trade Name _____

Composition _____

Specification _____

Generic Name _____

Batch / Lot Number _____

Use Temperature (minimum) _____

Use Temperature (maximum) _____

Propellant Fluid Exposure Time (field use) _____

Manufacturer

Name _____

Address _____

City _____

State _____

Country _____

Supplier

Name _____

Address _____

City _____

State _____

Country _____

Remarks _____

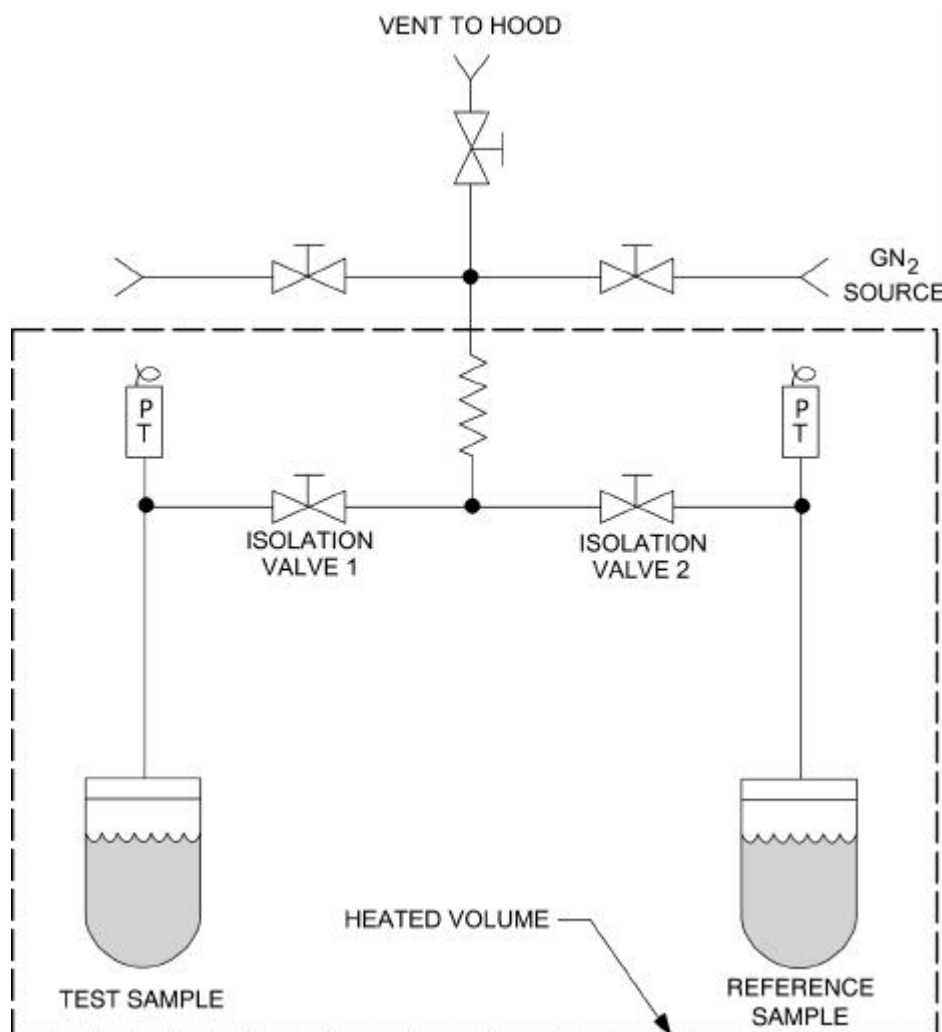


Figure 1 – Immersion test system

4.5.2.2 Immersion test

The immersion test shall be as follows:

- a) Place the test sample in the sample chamber and add sufficient liquid, approximately 25 millilitre (mL), to completely cover the test sample for the duration of the test. The addition of the test fluid must be performed to rigorously exclude water and carbon dioxide in the case of the propellant hydrazines and water in the case of nitrogen tetroxide. Contamination of the test fluid by these chemicals may give false indications of reactivity.
- b) Seal the sample chamber to the sample side of the test apparatus.
- c) Add sufficient test fluid to the reference chamber to obtain the same ullage as in the sample chamber.
- d) Seal the reference chamber to the reference side of the test apparatus.
- e) Activate the temperature and pressure monitoring devices.
- f) Heat both chambers at a rate of less than 2 °C per minute (min) until the test temperature, 70 °C minimum, is reached.
- g) Continue the test for 48 hours or until the pressure difference between the sample and reference transducers (PT) has exceeded the vapor pressure of the fluid plus 140 kilopascal (kPa).
- h) Allow the temperature to lower to ambient.

4.5.3 Posttest analysis

The posttest analysis shall be as follows:

- a) Perform a posttest analysis of the material and fluid to determine the extent of changes in the physical and chemical characteristics. The removal of the test fluid from the test system must be done in such a way as to exclude water and carbon dioxide contamination from the air.
- b) Measure changes in weight, dimension, texture, and color.
- c) Perform a comparison of the thermal properties of the material by differential scanning calorimetry for non-metals.
- d) Determine changes in the purity in the test fluid or residue by liquid chromatography, ion chromatography, atomic absorption spectrophotometer, inductively coupled plasma emission spectrometer, inductively coupled plasma/mass spectrometer, or gas chromatography/mass spectroscopy analyses.
- e) Compare the posttest fluid analysis with the appropriate fluid specification. A suitable test report form is shown in Figure 4 for a stainless steel sample. The posttest fluid analysis should be derived from the composition of the material being tested and have specific analytes appropriate to the composition.
- f) If requested, a graph of the volume of gas evolved versus time for both the test sample and reference sample may be provided.

4.6 Precision

Measurements shall be made to the following precision:

- a) Absolute pressure, ± 1 % of reading
- b) Temperature, ± 3 °C
- c) Sample dimensions, ± 5 % of the measurements
- d) Time, ± 5 minutes

Table 2 – Screening test report form

TEST SAMPLE MATERIAL DESCRIPTION

Test Conditions

Test Environment _____

Test Temperature _____

Test Duration _____

Test Results, Observations, and Comments

Pretest Weight: _____

Photograph Pretest Samples

Posttest Weight: _____

Material Characteristics

Component	Pretest Observations	Posttest Changes

Note(s): Pass _____ Fail _____

Posttest Photograph(s):

Table 3 – Posttest analysis test report form (sheet 1 of 2)

Test Sample Material Description

Geometric Surface Area _____

Test Conditions

Test Environment _____

Test Temperature _____

Test Duration _____

Reference Material: ISO 4954:1993

Geometric Surface Area _____

Test Results, Observations, and Comments

Pretest Weight _____

Posttest Weight _____

Average Gas Pressure

Sample	Reference

Table 3 – Posttest analysis test report form (sheet 2 of 2)

Material Characteristics

Component	Pretest Observations	Posttest Changes

Note(s):

Posttest Fluid Analysis

Analysis	Unit	Limit	Sample	Reference
Nonvolatile Residue	mg	0,1		
Bromide	mg	1,2		
Chloride	mg	0,5		
Chromium	µg	2,3		
Iron	µg	2,3		
Nickel	µg	2,3		

Notes: ND indicates that the results were less than the reporting limit.

Posttest Photograph(s):